Conversion of *Jatropha curcas* Oil to Ester Biolubricant Using Solid Catalyst Derived from Saltwater Clam Shell Waste (SCSW)

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Abstract—The discarded clam shell waste, fossil and edible oil as biolubricant feedstocks create environmental impacts and food chain dilemma, thus this work aims to circumvent these issues by using activated saltwater clam shell waste (SCSW) as solid catalyst for conversion of *Jatropha curcas* oil as non-edible sources to ester biolubricant. The characterization of solid catalyst was done by Differential Thermal Analysis-Thermo Gravimetric Analysis (DTA-TGA), X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Brunauer-Emmett-Teller (BET), Field Emission Scanning Electron Microscopy (FESEM) and Fourier Transformed Infrared Spectroscopy (FTIR) analysis. The calcined catalyst was used in the transesterification of *Jatropha* oil to methyl ester as the first step, and the second stage was involved the reaction of *Jatropha* methyl ester (JME) with trimethylolpropane (TMP) based on the various process parameters. The formated biolubricant was analyzed using the capillary column (DB-5HT) equipped Gas Chromatography (GC). The conversion results of *Jatropha* oil to ester biolubricant can be found nearly 96.66%, and the maximum distribution composition mainly contains 72.3% of triester (TE).

Keywords—Conversion, ester biolubricant, *Jatropha curcas* oil, solid catalyst.

I. INTRODUCTION

THE ineffective conversion of *Jatropha curcas* oil challenges problems for biolubricant processing. The biolubricant synthesis from edible sources like palm oil, soybean, etc. creates food versus fuel dilemma. Besides that, the major part of biolubricant manufacturing from the fossil fuel using homogeneous catalyst which also polluted the environment, difficulty separation and high cost, thus this work aims is to solve these issues by using saltwater clam shell waste (SCSW) for *Jatropha curcas* oil conversion to ester biolubricant.

Lubricant oil is used due to its decreasing ability of the frictions between two contacting surfaces by the separation with a film [1]. Fresh and oxidized lubricant compositions have no significant effect on the copper strip corrosion [2]. In the hydraulic system, the lubricant functions as power transmitter like chain in a mechanical driver [3]. In addition,

the lubricant oil also plays in the temperature control on auto motive system more roles, where it is able to absorb heat that produced during interaction between components of the operated system. The most commonly used raw material for lubricant synthesis is fossil (petroleum) oils, where it is readily accessible to fulfill the global market. Besides that, it has the longest activation life, and it is able to maintain the operating machine. Most modern lubricants are complex formulated products, it consist of 70%–90% mixed base oils with functional additives. These created formulation are directed to modify the natural properties such as cold stability, oxidation stability, hydrolytic stability and viscosity index to suit a specific application [4], [5]. Unfortunately, due to the great demands on the lubricants, there are growing concerns in searching for alternative lubricant feedstocks.

Nowadays, million tons of lubricants are dumped into environment through leakage from machine or vehicle and careless disposal methods, where some of these wastes are resistance to biodegradable. Thus, there are depressing needs in finding lubricants without leaving aside its principal properties and it can be compared with the based petroleum lubricant. Ester biolubricants are gaining interest due to its both renewable resources with independency on petroleum based oil, and its low greenhouse gas emission that help reduction of carbon dioxide (CO_2) effects on the environment. In this scenario, vegetables oils have gained popularity as economical lubricants over the last couple of decades that more efficient due to its excellent properties and ecological friendly [6], [7].

In the recent years, the *Jatropha curcas* oil as non-edible sources that available in Malaysia has been being investigating as a potential raw material for ester biolubricant synthesis. The commercial lubricant in market takes seriously on the stability, viscosity, lubrication and temperature range of lubricant oil characteristics, where it will be affected by the lifespan of the lubricant oil on the applied machine. Unfortunately, ester lubricant that derived from plant has limited properties, like poor low termo-oxidative and cold flow behavior. Chemical modification becomes favourable to overcome this problem via transesterification, epoxidation and hydrogenation process. In this case, transesterification of vegetables oil with polyhydric alcohol or polyols helps to minimize the limitation of biolubricant characteristics with the elimination of hydrogen atom from β-carbon of vegetables oil structure [8]. The transesterification takes place three phases with present of catalyst. Trimetylolpropane monoester (TMPME) and

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trimethylolpropane diester (TMPDE) become intermediate products, where the trimetylolpropane triester (TMPTE) is the main product of processing.

Catalyst plays the important role in the synthesis of biolubricant for obtaining better yield in the short time. Various catalysts have been being using in the production of biolubricant with the different kinds of vegetables oil as feedstocks. Homogenous catalysts such as inorganic acids and alkali have been being applied widely in the transesterification process either in biodiesel and biolubricant production. Previous researchers reports that the higher conversion of TMP triester can be found in 8 hours reaction time using calcium methoxide as catalyst [9], [10]. Otherwise, in the reality, it has been decreasing in these catalysts application due to a few problems, such as the catalysts cannot be regenerated or recovered after the reaction, and it also tend to produce toxic wastewater [11].

Heterogeneous catalyst such as metallic or metal oxide has wide potential to use it, where these catalysts can be easily recycled. Besides that, the metal oxide catalysts are much cheaper than enzyme or other biochemical catalysts. Currently, study on the transesterification process using solid base heterogeneous calayst such as calcium oxide that derived from mollusk shell has been interesting [12], [13]. Thus, the metal oxide from saltwater clam shell waste (SCSW) has been being developing as advantageous catalyst.

II. MATERIALS AND METHODS

A. Materials

The saltwater clam shell waste (SCSW) was collected from the sea food restaurants in Kuantan, Pahang, Malaysia. The *Jatropha curcas* oil was provided by the Bionas Sdn Bhd. Methanol, hydrocloric acid (HCl, 37%) and hexane (GC grade) were procured from the Sigma Aldrich Sdn Bhd. Trimethylolpropane (99%), N,O-bis (trimethylsilyl) trifluoroacetamide (BSTFA), ethyl acetate were purchased from the Chemmart Asia Sdn Bhd.

B. Methods

The collected saltwater clam shell waste (SCSW) was washed using clean water and dried at 100°C for overnight. Then, the solid catalyst was crushed and calcined in the furnace at 680°C for 2 hours. The activated catalyst was removed and kept in the desiccators to prevent air contact, and then it was characterized using FESEM, XRF, XRD, DTA-TGA BET, FESEM and FTIR analysis. In the same time, the acid-pre-treatment of the *Jatropha* seed oil was started with the esterification process. The oil was placed in two-necks flask equipped with magnetic stirrer and condenser. Then, the catalyst solution (a specified amount of HCl with 2% w/w of oil was dissolved in methanol with the molar ratio of 4:1 methanol to oil, and then it was poured into flask. The mixture was heated and stirred up to 60°C for about 1 hour. Then, the mixture was placed in the separatory funnel for excess methanol separation. The excess methanol was added with HCl, and the impurities moved to the top surface and removed. After acid pre-treatment process, base trans-esterification were carried out at the temperature of 60°C, 3%w/w amount of SCSW with the oil to methanol ratio was 1:4 for about 3 hours of reaction time. Then, the final product was poured in the separatory funnel. The catalyst with other debris accumulated in the bottom layer, meanwhile the upper layer known as *Jatropha* Methyl Ester (JME). The upper layer was collected and washed using warm water (50°C). The washing process was repeated until the water becomes clear.

The resulted JME was placed on the rotary evaporator for excess methanol removal by heating up to 750°C for 30 min. Next, the magnesium sulphate (2 g) was added and the sample was stirred for 30 min. The analysis of JME was performed using Gas Chromatography-Mass Spectrometry (GC-MS) for percentage composition of methyl ester determination. The sample was prepared in 2 ml vial; 0.02 ml of sample was mixed with 1.980 ml of hexane (GC- Grade). The analysis was conducted using GC equipped with DB-5HT column, 30 mm x 0.25 mm, i.d. of 0.10 μm (DB, United States). The injector and detector temperature were 380°C and 400°C. The initial oven temperature was set up at 100°C for 1 minute, and its initial holding time increased at 5 C /min up to 380°C for 25 min.

The batch system was set up for the *Jatropha* oil conversion, and the JME with TMP was put in a 250 ml three-necks round bottom flask equipped with thermometer, magnetic stirrer and reflux condenser. Prior to the reaction, amount of TMP was added and melted with constant stirrer in the flask at 60°C. After that, a calculated amount of JME (TMP to JME molar ratio 1:4) was placed into the reactor, and the temperature was raised to the desired reaction mixture post the catalyst addition. The reaction continued for a few hours at the vacuum condition (50 mmHg). The various temperature levels (90°C-130°C), amount of catalyst (0% w/w-4% w/w) and reaction times (1 hour to 5 hours) were run. The final products of transesterification process were TMP ester and methanol. After the reaction terminated, the final liquid product was handled by the vacuum filtration for solid catalyst and soap separation, and the product was cooled by room temperature. Furthermore, the ester biolubricant analysis was done by using Gas Chromatography (GC). The obtained TMP-ester in product based on the molecular mass provided the sample percentage composition. Approximately 0.03 ml of the samples was placed into the test tubes, and diluted with 1 ml of ethyl acetate and 0.5 ml N,O-Bis(trimethylsilyl) tri-fluoroacetamide (BSTFA). The mixture was wirld for a few minutes. The dissolved mixture and the vial were transferred into a 2 ml vial, and then it was heated at 400°C for 10 minutes. After cooling at room temperature, the sample was ready for GC-analysis. Then, the transesterification of JME with TMP involved three main stages for production of monoester (ME), diester (DE) and triester or tetraester (TE). Stoichiometry of the process required one mol of TMP reacted with three mols of JME. In order to avoid backwards process, the excess of JME was applied in this invention for 1:4 molar ratio of TMP. It was determined by the various temperatures,

catalyst loading and reaction times. The reaction conversion steps based on the four equations below:

$$ME + TMP \leftrightarrow ME + M \quad (1)$$

$$ME + ME \leftrightarrow DE + M \quad (2)$$

$$JME + DE \leftrightarrow TE + M \quad (3)$$
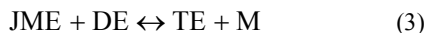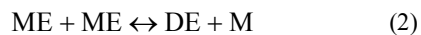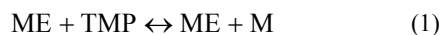
The overall reaction can be written, as in

$$3\,JME + TMP \leftrightarrow TE + 3M \quad (4)$$

The formulated biolubricant from the *Jatropha* oil conversion (TMP conversion) is calculated by the following formula (refer to (5)).

$$X_{TMP} = \left(1 - \frac{C_{TMP,P}}{C_{TMP,S}}\right) \times 100\ \% \quad (5)$$

where $C_{TMP,P}$ is the amount of TMP in product, $C_{TMP,S}$ is the amount of TMP added to the reaction system.

III. RESULTS AND DISCUSSION

A. X-Ray Fluorescence (XRF) of Saltwater Clam Shell Waste (SCSW)

The detailed chemical composition of the solid catalyst derived from SCSW was obtained from X-Ray Fluorescence (XRF) analysis, and it indicates a significant CaO content (86.25%) with small amount of other trace elements such as Na_2O (1.54%), SrO (0.29%), MgO (0.17%), SO_2 (0.15%), Cl (0.07%), Fe_2O_3 (0.06%), P_2O_2 (0.06%), SiO_2 (0.05%), K_2O (0.03%) and Al_2O_3 (0.03%). The used CaO has high potential for base catalyst of transesterification process due to its cheapness and non-toxic properties [14].Thus, the XRF shows that the solid catalyst from SCSW able to be used for biodiesel and ester biolubricant synthesis from the *Jatropha* oil.

B. Field Emission Scanning Electron Microscopy (FESEM) of Saltwater Clam Shell Waste (SCSW)

The morphology of catalyst from salt water clam shell waste (SCSW) was analyzed by FESEM. Fig. 1 (a) shows that the surface of catalyst before activated is rough and disordered surface with low porosity structure compared after activation process. Otherwise, Fig. 1 (b) illustrates a relatively smooth with spherical particles of porous structure that confirming the thermal decomposition.

C. X-Ray Diffraction (XRD) of Saltwater Clam Shell Waste (SCSW)

The XRD peaks were shown broad in Fig. 2. Fig. 2 (a) indicates mainly $CaCO_3$ structure form before calcination. The peak is illustrated at 26.22^0 until 52.99^0. Meanwhile, after calcination is shown in Fig. 2 (b), whereas $CaCO_3$ structure is lost, but CaO appears in the peak. The peaks are 32.296^0, 37.442^0, 53.954^0 and 64.16^0.
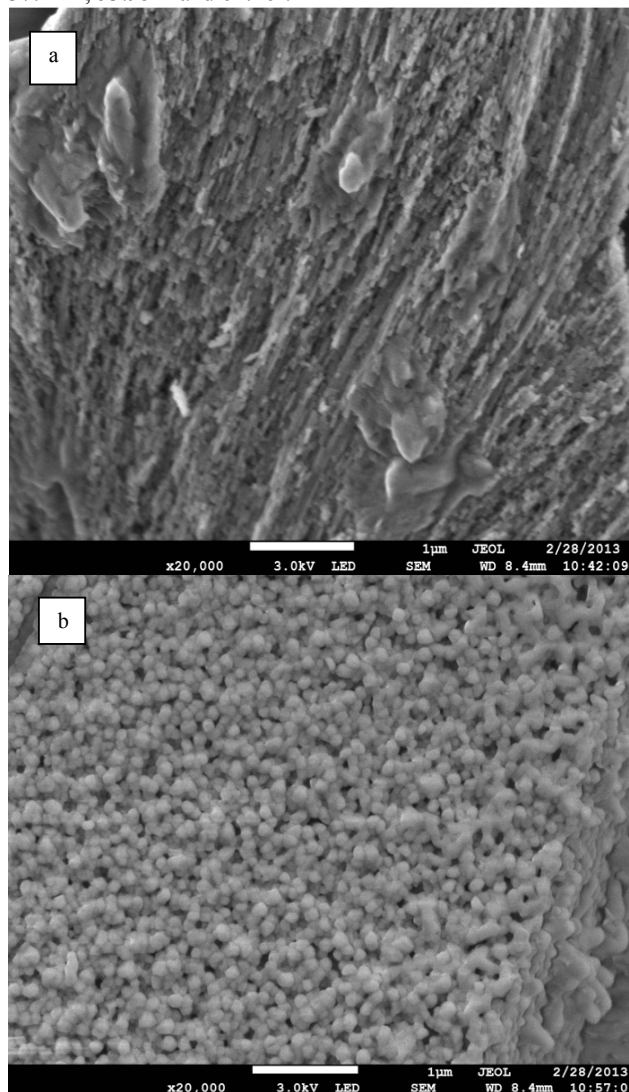

Fig. 1 FESEM micrograph of saltwater clam shell waste (SCSW) at 20,000 x, before activation (a), after activation (b)
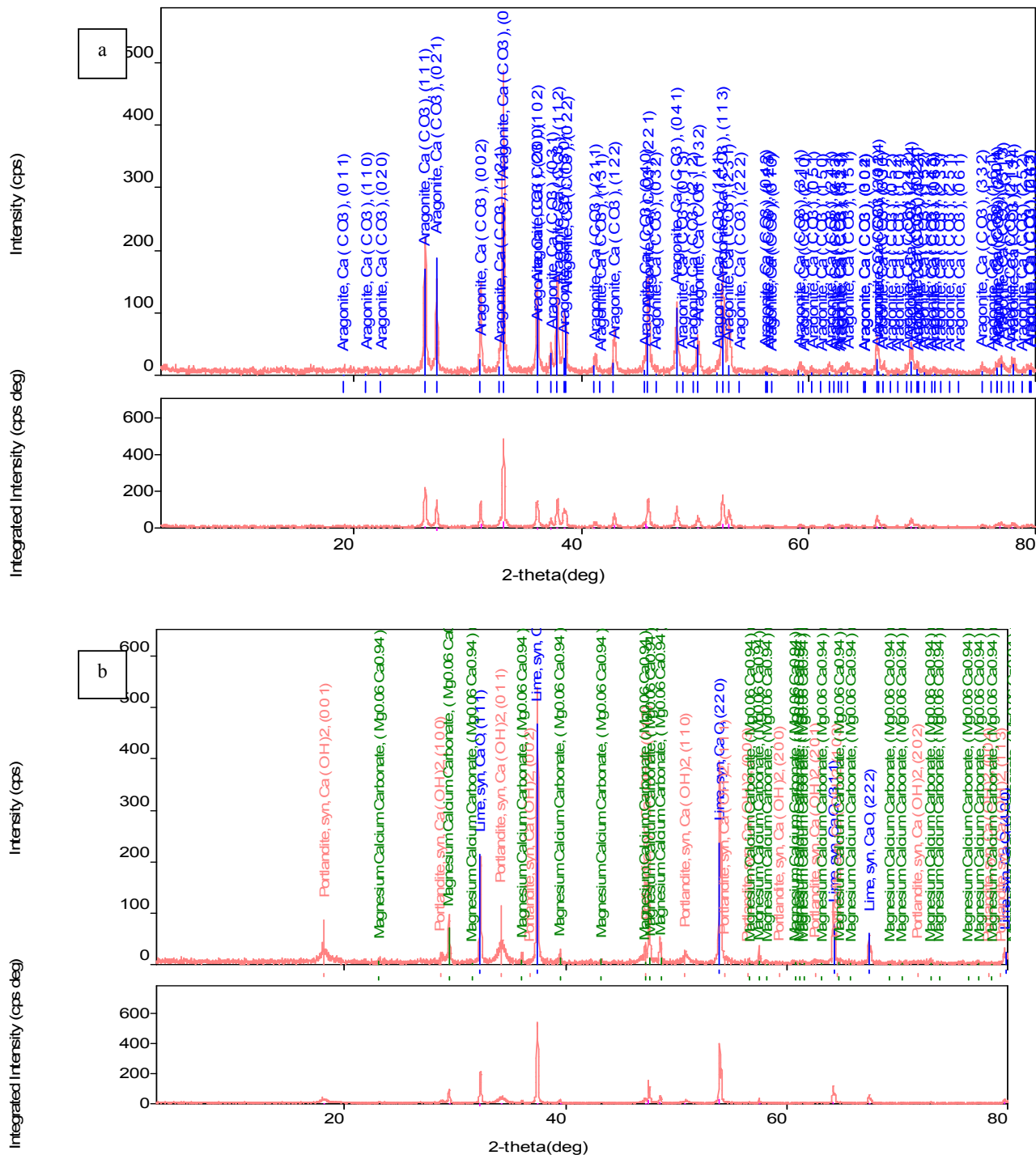
Fig. 2 XRD of saltwater clam shell waste (SCSW), before activation (a), after activation (b)

D. Differential Thermal Analysis and Thermogravimetric Analysis (DTA-TGA) of Saltwater Clam Shell Waste (SCSW)

The saltwater clam shell waste was analyzed using DTA-TGA for the suitable calcination temperature. The temperature range of 610°C–760°C was operated for calcium oxide synthesis from the saltwater clam shell. The saltwater clamp shell waste (SCSW) requires above 610°C for calci-nation process where the clam shells containing $CaCO_3$ are generally known as a main based product, and it needs to a high temperature to transform $CaCO_3$ to CaO which can be determined by the TGA and DTA analysis. The analysis has

shown the resulted CaO by the selected optimum temperature nearly 680°C (Fig. 3).
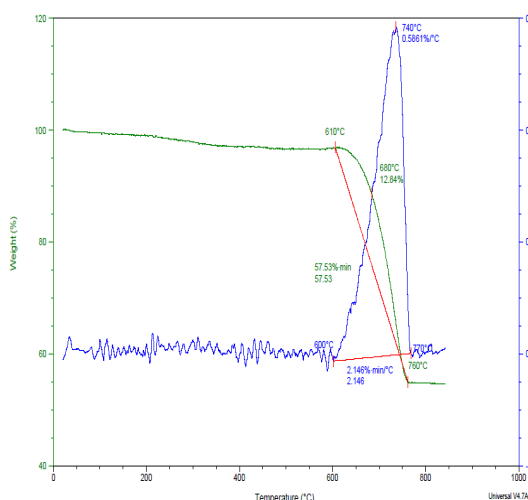

Fig. 3 DTA-TGA analysis of of saltwater clam shell waste (SCSW)

E. Brunauer-Emmett-Teller (BET) of Saltwater Clam Shell Waste (SCSW)

The BET analysis of saltwater clam shell waste (SCSW) was conducted by temperature of 680°C for 2 hours, and it shows the results in Table I. Table I gives the pore size of catalyst before calcinations is 183.7892 Å, and after calcination, it shifts to 312.527 Å. This comparison proves that the calcination process plays the catalyst activity more roles. The BET surface area of the calcined catalyst is 2.1290 m^2/g. Since the pore volume of SCSW is 0.005986 cm^3/g that less than 0.03 cm^3/g, the material appears to have non porous structure.

TABLE I
BET ANALYSIS OF SALTWATER CLAM SHELL WASTE (SCSW)

Source of Catalyst	Saltwater clam shell waste
Calcinations Temperature (°C) and time (h)	680 and 2
BET Surface Area (m^2/g)	2.1290
Pore Volume (cm^3/g)	0.005986

F. Fourier Transformed Infrared Spectroscopy (FTIR) of Saltwater Clam Shell Waste (SCSW)

The FTIR spectra of both saltwater clam shell waste (SCSW) shown in Fig. 4. The catalysts before and after activation occur the major peak of $CaCO_3$. The peak of non-activated catalyst gives 1 471, 856 cm^{-1} and 712 cm^{-1}. Meanwhile, after calcination the major bands are obtained at 1471.05, 876.42 cm^{-1} and 558.07 cm^{-1}. After calcination, the molecules move to the higher energy with the reduction of functional group.
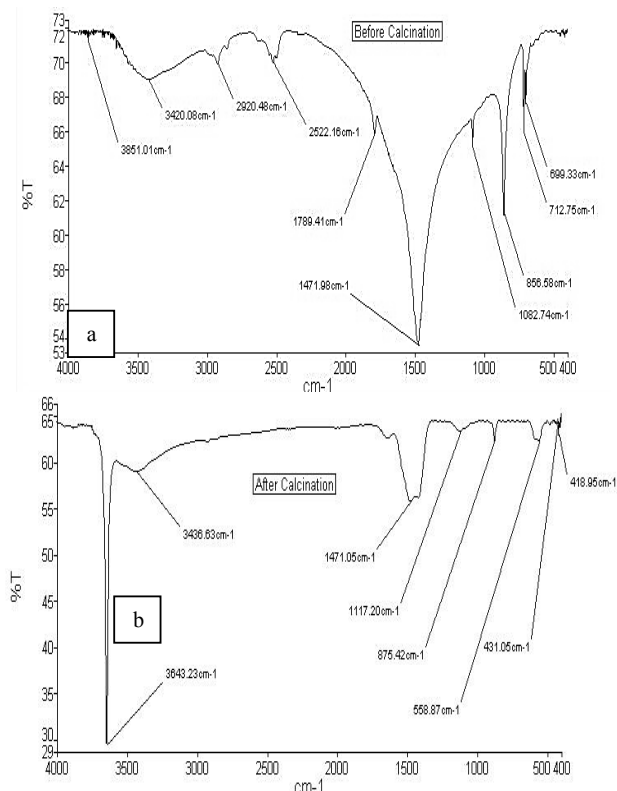

Fig. 4 FTIR analysis of saltwater clam shell waste (SCSW), before activation (a), after activation (b)

G. Analysis of Jatropha Methyl Ester (JME) Using Gas Chromatography (GC)

The obtained *Jatropha* methyl ester (JME) as feedstock for the second stage processing of ester biolubricant was analyzed using GC and the results are shown in the Fig. 5 and Table II. Fig. 5 shows the significant presence of methyl ester from JME using activated saltwater clam shell waste (SCSW). The identification of the peak can be performed to determine the methyl ester. Meanwhile, Table II approves the fatty acid methyl ester content from *Jatropha curcas* oil, which are mainly methyl oleate (37.75%) and methyllinoleate (36.70%). These values were higher than the Net Coconut Oil (NCO) [15]. The NCO contains the methyl oleate and linoleate of 6.41% and 5.41%.

TABLE II
JATROPHA METHYL ESTER (JME) COMPOSITION

| Compounds	Retention time (min)	Percentage area (%)
Methyl Myristate	25.246	0.05
Methyl Palmitate	28.868-29.539	16.85
Methyl Margarate	31.270	0.10
Methyl Linoleate	32.735	36.70
Methyl Oleate	32.956	37.75
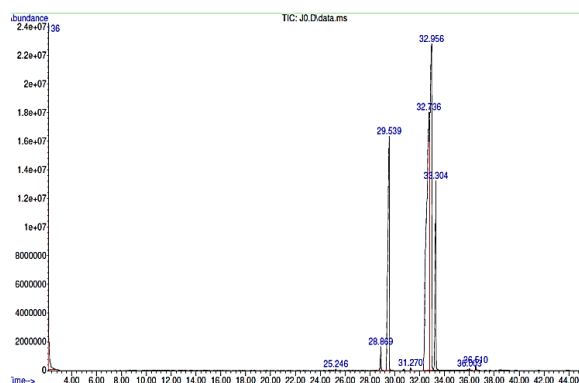
Fig. 5 GC analysis of FAME from *Jatropha curcas* oil

H. Effect of Temperature on the Jatropha Oil Conversion

The effect of temperature on the *Jatropha* oil conversion to ester biolubricant plays the important role. The results can be shown in Fig. 6. The curve indicates conversion of TMP and its composition simultaneously. The highest conversion of TMP to biolubricant ester is 96.66%, and 72.3% of TE composition, it is found at 110°C. The low temperature tends to reversible reaction; it has been approved by using rubber seeds oil as biolubricant feedstock. On the other hands, it is reflected that at the higher temperature, colour of product is turn to dark due to oxidation of oil [16]. Thus, the higher temperature is not only higher energy consumption, but also may disturb the appearance of product.
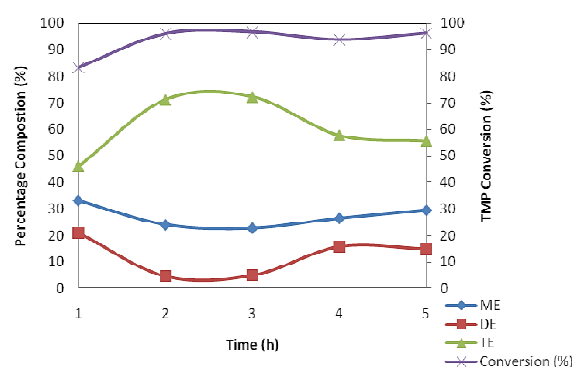
I. Effect of Catalyst Amount on the Jatropha Oil Conversion

The various catalyst loading were given for the *Jatropha* oil conversion to ester biolubricant. Fig. 7 shows 3% w/w of the required optimum amount of catalyst. Above the optimum level of catalyst, it is reflected the decreased conversion, and it could be the increased soap formation. Besides that, if the required catalyst is rich, increases the solid content on the product. It causes the final conversion disturbance [17].
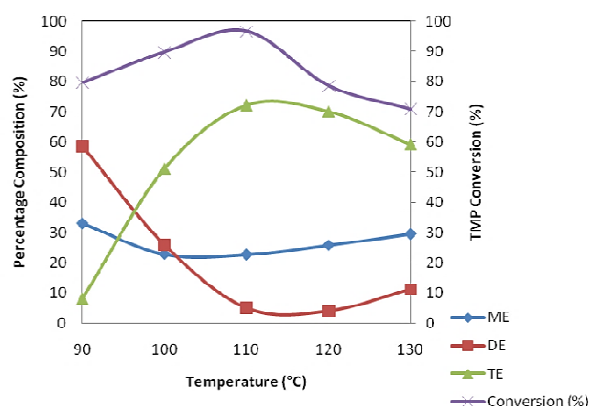
Fig. 6 Effect of temperature on the *Jatropha* oil conversion (p=50 mbar, catalyst =3% w/w, TMP: JME=1:4, t= 3 h)
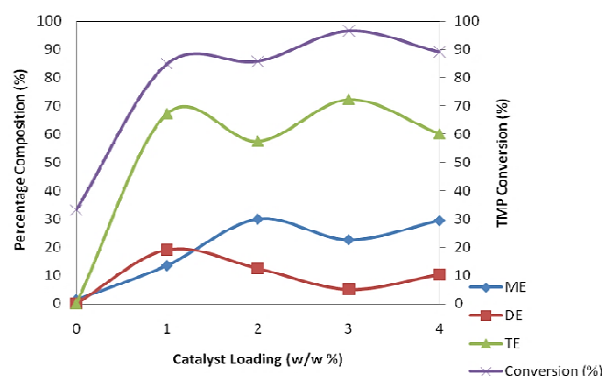
Fig. 7 Effect of catalyst loading on the *Jatropha* oil conversion (p=50 mbar, T=110 °C, TMP: JME=1:4, t=3 h)

J. Effect of Reaction Time on the Jatropha Oil Conversion

The reaction time was investigated from 1 hour to 5 hours for *Jatropha* oil conversion. Fig. 8 reflects the increase of conversion during 1 hour to 3 hours, but the slightly decreased conversion takes place after 3 hours. The reaction time is also influenced by the types of used catalysts [18]. If the reaction time is longer, results the decreased composition of TMP Triester (TMPTE). It can be predicted due to hydrolyzation phenomenon, and the reaction tends toward TMP Monoester (TMPME) and TMP Diester (TMPDE).

Fig. 8 Effect of reaction time on the *Jatropha* oil conversion (p=50 mbar, T=110 °C, catalyst =3% w/w, TMP: JME=1:4)

IV. CONCLUSION

Conversion of *Jatropha curcas* oil as feedstock to ester biolubricant can be achieved using solid catalyst derived from saltwater clam shell waste (SCSW). The activated solid catalyst reflects the higher surface area compared non-activated saltwater clam shell waste. The two-stages processing for *Jatropha* oil conversion using heteregenous catalyst and trimetylolpropane triester (TMPTE) results the ester biolubricant about 96.66% by the optimum temperature of 110°C, catalyst loading of 3% w/w and reaction time of 3 hours. The maximum composition distribution of TMP ester comprises almost 22.7% of ME, 5% of DE and 72.3% of TE. This obtained operation condition, two-stages treatment and solid catalyst could be developed for biolubricant conversion

and quality improvement via various process parameters, additives, methods and heteregenous catalysts.

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